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# **IMPORTANT NOTES FOR CALCIUM ISE SYSTEM**

## Calibration and Cleaning of Calcium Selective Ion Measurement System For Waste Water Calcium Analysis

- All calibration solutions and Process Grab Sample should be calibrated and tested at identical temperatures to the process temperature to minimize errors due to temperature effects. The actual temperature of the process solution (and thereby the calibrating solutions as well) is not as critical as the fact that they are calibrated at the same temperature. To optimize calcium sensor  $(Ca^{+2})$  lifetime, however, measurement should be performed as close to 25 degrees Celsius (room temperature process solutions) as possible.
- Calibration Point 1 (always the lower concentration value) and Calibration Point 2 (always the higher concentration value) determines the response of the 54e Analyzer to a given Calcium Ion Selective Sensor (AB 6440 & AB 8440)
- The One Point Re-Calibration Standard (usually a process grab sample or one of the two calcium standard solution) is always performed by using the standardize option from the Calibrate Main Menu. A one-point calibration should never be performed by using the 2-point calibration option from the Calibrate Main Menu.
  - The calibration solutions should be kept clean and out of direct sunlight and/or other high-energy radiation sources for any extended period of time. New sensors should be conditioned in calcium standard solution for 3 5 minutes before beginning calibration procedure as described in this quick calibration guide.
- The acceptable pH range of the AB 6440 & AB 8440 Calcium Sensor is 2.5 11.0. Since calcium ion activity is affected by pH, the pH should be stable to ensure meaningful and reproducible results. Slightly acidic media are recommended whenever possible.
- To enter custom ion menu, hold F3 for 5-7 seconds, then enter secret code of 20000 into system.

### The following steps are required for an installation of a NEW calcium sensor:

- 1) Enter the nominal ISO Voltage (- 81 mV) and Slope (+ 31.00 mV per decade) as given on the proceeding page.
- 2) Perform a two-point calibration to empirically determine slope. See attached procedures for 2-point calibration.
- 3) Perform a one-point calibration (standardize) at the defined (expected) measurement value. See the proceeding section one point calibration for further details on use of this calibration function.
- 4) Place sensor into process and allow to it to find equilibrium. At this point, another one-point calibration (standardize) may be required. This should be performed WITHOUT taking the sensor out of service. See below for how to perform the standardize one-point offset calibration without removing the ISE sensor from service.
- 5) To account for any differences between the ionic strength and interfering ions between calibration and measured solution, a grab sample should be taken and analyzed by an alternate analysis system, and the online Calcium system adjusted to read the analyzed value. The sensor should be left in process and this grab sample (standardize) calibration performed as may be required, unless the sensor seems to be losing sensitivity or giving erratic readings.

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# MENU for ROSEMOUNT 54e CALCIUM ANALYZER

Main Sub-Menu	Calibrate Main Menu
Calibrate Sub-Menu	2-Point Calibration
Low	Calibration Point 1
High	Calibration Point 2
Enabled	Hold Option for Outputs 1 & 2: Read Rosemount 54e-pH/ORP Manual for Details
Calibrate Sub-Menu	Standardize
Variable	Standardize Concentration: Value = Grab Sample, Calibration Solution 1 or 2:
	See calibration instructions for details on this function; a.k.a. 1-point calibration
Calibrate Sub-Menu	Adjust Temp
User Defined	Manual Adjustment of temperature, not required for most installations
Calibrate Sub-Menu	Temp Compensation
Setting / Value	FUNCTION / PARAMETER
Manual 25°C	Temperature Compensation
Main Sub-Menu	Diagnostie Variables
	Diagnostic Variables
Defined by 54e	Displays all currently defined variables in 54e Memory
Main Sub-Menu	Program
Program Sub-Menu	Alarm Setpoints
Rosemount Default	Variable alarm setpoints for 3 @ relays
0.000 ppm & 999.9 ppm	Any user defined value within measurement output range is acceptable
Program Sub-Menu	Output Setpoints
4mA= 0.000 ppm	4-20mA Output 1 – Process Calcium ISE
20mA = 1000  ppm	These are only the factory defaults. These values can be altered.
90 seconds	Time Average of Process Output
$4mA = 5^{\circ}C$	4-20mA Output 2 – Temperature
$20\text{mA} = 50^{\circ}\text{C}$	These are only the factory defaults. These values can be altered.
20 seconds	Time Average of Temperature Output
Program Sub-Menu	Diagnostics
Disabled	Disabled diagnostics required to make Calcium ISE measurement run properly
	(Factory Defined – DO NOT MODIFY)
Program Sub-Menu	Simulated Test
Rosemount Default	Read Rosemount 54e-pH/ORP Manual for Details
Program Sub-Menu	Configure
ASTI Programmed	Passcode to enter Configure Function is 974. Various parameters in this function may affect
Passcode for Configure	output and display of Analyzer (Optional)
Ca <sup>+2</sup>	Selective Ion Measurement Parameter (Factory Defined – DO NOT MODIFY)
ppm	Display units for $Ca^{+2}$ Measurement (Defined in Program-Configure Menu)
°C	Display units for Temperature Measurement (Defined in Program-Configure Menu)
-999 to + 999	ISE Input Range in mV's – Rosemount Defined
-777 IU T 777	
Custom Ion Curve	Press F3 for 5-7 seconds, enter Passcode of 20000
40.10 grams per mol	IONIC WEIGHT (Defined by Selective Ion Measurement – DO NOT MODIFY)
5 ppm (3.903936)	ISOPOTENTIAL CONCENTRATION (Factory Defined – DO NOT MODIFY)
+ 31.00 mV per decade	DEFAULT SLOPE (Will be changed when 2-point calibration is performed)
- 81 mV	ISOPOTENTIAL VOLTAGE (Will be changed when 2-point calibration is performed)
01 111 7	is of order that is obtained (it in be changed when 2 point canoration is performed)

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# Calcium (Ca<sup>+2</sup>) Probe Two Point Calibration

This calibration method should not need to be performed frequently. All new calcium sensors should be calibrated using a 2-point calibration first. Subsequent calibrations can be made using only the standardize option from the Calibrate Main Menu. This calibration determines the sensitivity or slope of each sensor, which is then stored in the 54e transmitter. A "standardize" (a.k.a. 1-point) calibration must be performed after every 2-point calibration. Details for the 1-point calibration are given in the proceeding page. MAKE SURE THAT THE "HOLD" FEATURE IS ON BEFORE STARTING ANY CALIBRATION. READ THE ROSEMOUNT 54E-PH/ORP MANUAL FOR FURTHER DETAILS ABOUT THE HOLD FEATURE.

#### Set-up requirements:

- Two 250 mL GLASS OR PLASTIC BEAKERS.
- Low Calcium Standard Solution
- High Calcium Standard Solution

Standard calcium solutions are provided by ASTI. These standard solutions are re-ordered by indicating the special part numbers or notes (if any) that appear on the calibrating solution bottles. Do not dilute or in any other way modify these standardization solutions. Keep calcium solutions out of direct sunlight or any other radiation source.

Follow the on-screen directions in 2-point calibration submenu in the 54e Analyzer. Calibration Points 1 & 2 have been preprogrammed into your instrument. The low standard solution will always be Calibration Point 1 & the high standard solution will always be Calibration Point 2. Do not change the preprogrammed values for Calibration Point 1 & 2.

#### **Important Notes about Calibration:**

- Fill a 250 mL GLASS beaker with enough standardization solution such that the entire tip of the calcium sensor will be submersed
- Please read the attached sheet on the cleaning procedure for this ion selective sensor. The cleaning procedure should usually be performed when transferring a sensor in or out of solution. Sensors can also be cleaned before being placed into different concentration standardization solutions and/or grab sample solutions. Cleaning is only required if fouling appears on the ISE membrane or on the reference junction. If no contamination is apparent, then the simple rinsing procedure given below can be used.
- Thoroughly rinse the sensors with DI water and gently blot dry with a clean lint free towel. Be careful not to scratch or damage the sensitive organic calcium ion selective membrane
- Allow a minimum of 3 5 minutes for the sensor to stabilize once it has been removed from the process and placed into the low standard solution.
- Allow a minimum of 3 5 minutes for the sensor to stabilize between the low and high Calibration Solutions.

#### Key Sequence for 2-Point Calibration

- 1. Press any Function Key
- 2. Highlight Calibrate and Enter Function
- 3. Select Hold Mode to On and Continue
- 4. Select 2-Point Calibration and Enter Function
- 5. Calcium sensor should be in low standard solution already cleaned and conditioned. Press Continue. Instrument will display that Calibration Point 1 is stabilizing. The instrument will take about 20 seconds to stabilize and determine mV value for the first calibration point.
- 6. After Cal Point 1 has stabilized select edit and input Low Cal value into instrument. This value must be entered even if it already is correct on the display. After low Cal value has been entered into Analyzer, press save to continue to second calibration point.
- 7. Calcium sensor should be in high standard solution already cleaned and conditioned. Press Continue. Instrument will display that Calibration Point 2 is stabilizing. The instrument will take about 20 seconds to stabilize and determine mV value for the second calibration point.
- 8. After Cal Point 2 has stabilized select edit and input High Cal Value into instrument. This value must be entered even if it already is correct on the display. After high cal value has been entered into Analyzer, press save.

THE ROSEMOUNT ANALYZER AT THIS POINT SHOULD DISPLAY "2-POINT CALIBRATION DONE". ONLY IF THIS MESSAGE APPEARS HAS THE TWO-POINT CALIBRATION BEEN SUCCESFULLY PERFORMED, OTHERWISE REPEATED STEPS AS OUTLINED ABOVE UNTIL THE ANALYZER ACCEPTS THE TWO-POINT CALIBRATION.

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# Single Point Calibration -

This is the correct method to Adjust for Sensor Drift

### **Calcium Sensor One Point Calibration**

- Only use the "Standardize" function from the Calibrate Menu. Any other method will destroy the previous 2-point calibration
- Can be performed DAILY if required.

When the calcium system has been calibrated by the 2-point method it can then be used in the "single point" calibration mode (on a daily basis if required). MAKE SURE THAT THE "HOLD" FEATURE IS ON BEFORE STARTING ANY CALIBRATION. READ THE ROSEMOUNT 54E-PH/ORP MANUAL FOR FURTHER DETAILS ABOUT THE HOLD FEATURE.

#### Set-up requirements:

- Two 250 mL GLASS OR PLASTIC BEAKERS
- Low Calcium Standard Solution or
- High Calcium Standard Solution or
- Process Grab Sample Solution

Follow the on-screen directions in the "Standardize" submenu of the Calibration Menu in the 54e Analyzer. No value has been preprogrammed into your instrument for the standardization concentration. Both the low and high standardization solutions can be used to perform the "1-point" calibration performed by the standardize function. In addition, grab samples can be taken from the process and analyzed by an alternate method for ion concentration. The separately determined concentration of the process sample can then be entered into the standardize menu. In this way, the calcium sensor can be standardized without ever having to remove the sensor from the process line.

### Important Notes about 1-point "Standardize" Calibration:

- Fill a 250 mL GLASS beaker with enough standardization solution such that the entire tip of the calcium sensor will be submersed
- Read the attached sheet on the cleaning procedure for this ion selective sensor. The cleaning procedure should usually be performed when transferring a sensor in or out of solution. Sensors can also be cleaned before being placed into different concentration standardization solutions and/or grab sample solutions. Cleaning is only required if fouling appears on the ISE membrane or on the reference junction. If no contamination is apparent, then the simple rinsing procedure given below can be used.
- Thoroughly rinse the sensors with DI water and gently blot dry with a clean paper towel. Be careful not to scratch or damage the sensitive organic calcium ion selective membrane
- Allow a minimum of 3 5 minutes for the sensor to stabilize once it has been removed from the tank and placed into either the low or high standard solution.

#### Key Sequence for 1-Point Calibration

- 1. Press any Function Key
- 2. Highlight Calibrate and Enter Function
- 3. Select Hold Mode to On and Continue
- 4. Select Standardize and Enter Function
- 5. Calcium sensor should be in standard solution already cleaned and conditioned. Allow reading to stabilize. Press Edit. Enter low calibration standard value into Analyzer and press save. The one point calibration is now complete. The reading on the display should be same as the entered standardize value. Alternatively, the high calibration solution can be used for 1-point calibration if this is expected measurement value. If a grab sample is used, allow the sensor to find a stable reading in process. Enter the analyzed value of the process solution for the calcium concentration. The sensor should be left in service while this standardize calibration is performed.

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# Cleaning and Maintenance of ASTI "Ca<sup>+2</sup>" Probe

Before a major 2-point calibration the sensor may need to be cleaned each time. The frequency of cleaning will depend on the quality of the process water and the build up of process reagents on the probe tip.

### Note:

Any noticeable deposits on the tip of the sensor will result in a less accurate calibration and measurement.

#### **CLEANING:**

- 1. Thoroughly rinse the sensor tip with DI water. Gently blot the sensor tip dry.
- 2. Scrape the entire Kynar reference area clean with a sharp blade or Stanley knife. This reference is solid Kynar and cannot be damaged. Do <u>not</u> scratch the membrane.
- 3. Once this has been achieved the entire tip can be soaked in either the low or high standardization solution. After allowing sufficient time for conditioning; proceed to perform a 2-point or 1-point calibration.
- 4. The standardization solutions can always be used as conditioning solution for extended storage. Do not allow sensor to be exposed to air for prolonged periods of time. Always store sensor in standardization solution when not in service in process. For long-term storage, a standard solution should be placed into sensor protective cap. The cap should be sealed onto sensor body with Teflon tape.

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### **Miscellaneous**

- The decimal place can be moved in any screen of the 54e Analyzer by placing the cursor over the decimal place and using the up and down arrows to move the decimal point to any position.
- Do not to allow air bubbles to get trapped near the calcium ion selective organic membrane. This is will cause erroneous readings and drift.
- The calcium sensor is comprised of a high-impedance organic membrane system. Care should be taken not to move or touch the cable once a value is being stabilized. Touching the sensor cable can cause a noisy signal that may result in erroneous values and calibrations.
- Please see the hook-up schematic found in the AB 6440 & 8440 calcium sensor shipping box, together with the specification sheet. Be sure to check whether that the jumper in the instrument is set for an external preamplifier is a preamplifier has been embedded into the sensor (preamplifier in sensor setting) or for an internal preamplifier (using the preamplifier built into the 54e) if the sensor does not have any preamplifier (100 Ohm TC only).